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4-Bromo-1-[2,6-dichloro-4-(trifluoromethyl)-phenyl]-5-[(2-furyl)methyleneamino]-1*H*-pyrazole-3-carbonitrile

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The title compound, $C_{16}H_6BrCl_2F_3N_4O$, is an imine with an overall Y-shape. The dihedral angles between the pyrazole ring and the benzene and furan ring planes are 88.6 (2) and 65.5 (2)°, respectively.

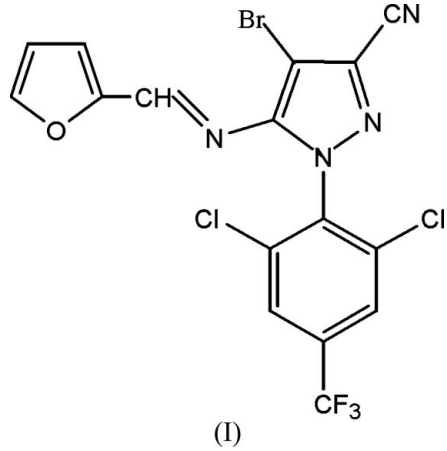
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.047
wR factor = 0.136
Data-to-parameter ratio = 13.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The title compound, (I) (Fig. 1), is similar to the very effective insecticides used to treat animals such as cows and sheep (Philippe, 1997, 2000) and its structure is reported here. The molecule of (I) contains three essentially planar rings. The dihedral angles between the pyrazole ring (C8–C10/N1/N2) and the benzene (C2–C7) and furan (C13–C16/O1) ring planes are 88.6 (2) and 65.5 (2)°, respectively. There are no π–π stacking interactions in (I).



(I)

Experimental

Following the method of Zhong *et al.* (2005), using 5-amino-3-cyano-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]pyrazole (2.5 mmol), followed by reaction with furaldehyde (2.5 mmol) and concentrated hydrochloric acid (2 ml) in anhydrous ethanol (5 ml), we obtained 1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-3-cyano-5-[(2-furyl)methyleneamino]1*H*-pyrazole, which was then reacted with *N*--bromosuccinimide (1.5 mmol) (Philippe, 2000) in acetonitrile (6 ml) at room temperature. After being stirred a few minutes, the reaction was monitored by thin-layer chromatography until the consumption of the starting materials was complete. Finally, the reaction mixture was evaporated under reduced pressure to provide the required crude product, which was then partitioned between dichloromethane and water; separating and drying the organic phase and evaporating it under reduced pressure gave the title compound (87.4% yield). Colourless single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an anhydrous ethanol–acetone (2:1 *v*/*v*) solution of (I) (m.p. 432–433 K).

Crystal data

$C_{16}H_6BrCl_2F_3N_4O$
M_r = 478.06
Monoclinic, $P2_1/c$
a = 9.561 (2) Å
b = 22.927 (5) Å
c = 8.4322 (19) Å
β = 96.220 (4)°
V = 1837.6 (7) Å^3
Z = 4
D_x = 1.728 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 2.57 mm^{-1}
T = 298 (2) K
Block, colorless
0.49 × 0.37 × 0.20 mm

Data collection

Bruker APEX CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.366, T_{max} = 0.627
9676 measured reflections
3302 independent reflections
2519 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 25.3°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.136
S = 1.04
3302 reflections
244 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0669P)^2 + 1.8001P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.64 e Å^{-3}
$\Delta\rho_{min}$ = −0.67 e Å^{-3}

All H atoms were initially located in a difference Fourier map and were relocated in idealized locations (C—H = 0.93 Å) and refined as riding, with $U_{iso}(H) = 1.2_{eq}(C)$. The high displacement parameters for atoms F1, F2 and F3 indicate either large torsional motion or rotational disorder of the trifluoromethyl group. However, attempts to represent the –CF_3 group using a disorder model were unsuccessful.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

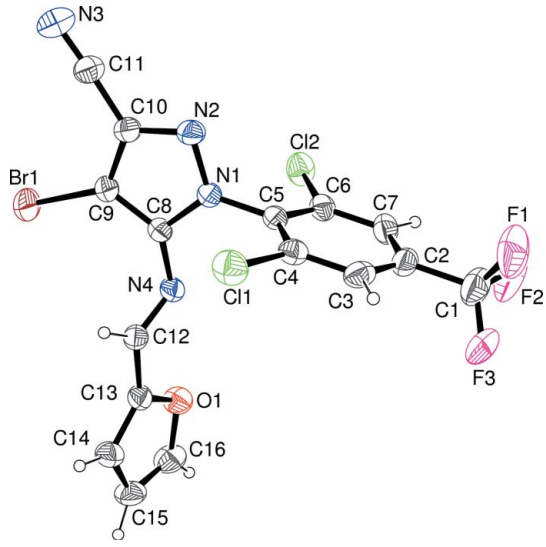


Figure 1
The molecular structure of (I), showing 50% displacement ellipsoids (arbitrary spheres for the H atoms).

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